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(तीसरा पुनरीक्षण)

Indian Standard

DEXTROSE MONOHYDRATE — SPECIFICATION

(*Third Revision*)

UDC 664·162

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

April 1992

Price Group 5

AMENDMENT NO. 1 JULY 2002
TO
IS 874 : 1992 DEXTROSE MONOHYDRATE — SPECIFICATION

(Third Revision)

(*Page* 2, *clause* 5.2.2) — Substitute the following for the existing clause

' **5.2.2** *BIS Certification Marking*

The product may also be marked with the Standard Mark

5.2.2.1 The use of the Standard Mark is governed by the provisions of *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made thereunder The details of conditions under which the licence for the use of Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards '

(FAD 2)

Reprography Unit BIS New Delhi India

AMENDMENT NO. 2 JUNE 2011
TO
IS 874 : 1992 DEXTROSE MONOHYDRATE — SPECIFICATION

(Third Revision)

[*Page* 2, *clause* **5.2.1**(e)] — Substitute 'Net quantity' *for* 'Net mass'.

[*Page* 2, *clause* **5.2.1**(f)] — Substitute the following for the existing:

> 'Any other requirements as given under the *Standards of Weights and Measures (Packaged Commodities) Rules*, 1977 and the *Prevention of Food Adulteration Act*, 1955 and the Rules framed thereunder.'

(FAD 2)

Reprography Unit, BIS, New Delhi, Indi

Edible Starches and Starch Products Sectional Committee, FADC 17

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Edible Starches and Starch Products Sectional Committee had been approved by the Food and Agriculture Division Council

This standard was first published in 1956 Its first revision was brought out in 1965 in which the limit for arsenic was changed, the limits for lead and copper substituted for heavy metals and the requirements for chloride, sulphates, less soluble sugars, solubility and dextrins deleted

In this revision additional requirement for purity, specific volume, total solids content and other metallic impurities, namely, tin, zinc, mercury and cadmium have been included

This standard covers requirements for dextrose monohydrate suitable for use in the food processing industry The requirements for dextrose monohydrate used for pharmaceutical purposes are stipulated in the (IP) Indian Pharmacopoeia and are, therefore, not covered in the provisions of this specification.

In the preparation of this standard due consideration has been given to the provisions of the *Prevention of Food Adulteration Act, 1954* and the Rules framed thereunder and the Standards of Weights and Measures (Packaged Commodities) Rules, 1977 However, this standard is subject to the restrictions imposed under these, wherever, applicable

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard

Indian Standard

DEXTROSE MONOHYDRATE — SPECIFICATION

(Third Revision)

1 SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for dextrose monohydrate suitable for use in the food processing industry.

1.2 This standard does not apply to dextrose monohydrate meant for pharmaceutical purposes.

2 REFERENCES

The Indian Standards listed in Annex A are necessary adjuncts to this standard

3 TERMINOLOGY

3.0 For the purpose of this standard, the following definitions shall apply.

3.1 Dextrose Monohydrate

A dextro-rotatory monosaccharide having the formula $C_6H_{12}O_6 \cdot H_2O$.

3.2 Optical Rotation

The angle through which the plane of polarization is turned when a layer of the liquid substance, one decimetre in thickness and at a temperature of 25°C, is examined under sodium light.

4 REQUIREMENTS

4.1 Description

The material shall be purified and crystallized *d*-glucose containing one molecule of water of crystallization. It shall be white or light cream in colour The material shall be a crystalline or granular, odourless powder readily soluble in water with a characteristic sweet taste free from foreign flavour.

NOTE — The appearance, taste and odour shall be determined by sensory tests

4.2 Freedom from Colouring Matter

The material shall be free from any added colouring matter.

4.3 Freedom from Foreign Matter

The material shall be free from dirt, other extraneous matter, insects, rodent of other contamination.

4.4 Loss on Drying

When tested by the method prescribed in Annex B the loss on drying shall be not less than 7.5 percent and not more than 9.5 percent by mass

4.5 Specific Rotation

The specific rotation of solution containing one gram of the material, previously dried at 105°C for 6 hours, with 0.02 ml of ammonium hydroxide solution in each 10 ml of water shall be not less than +52.5° and not more than +53.3°, when tested by the method prescribed in Annex C

4.6 Specific Volume

The specific volume of the material shall not be more than 160 ml/100 g when tested by the method given in Annex D

4.7 The material shall also comply with the requirements given in Table 1

Table 1 Requirements for Dextrose Monohydrate

Sl No.	Characteristic	Requirement	Method of Test, Ref to	
			Annex of This Standard	Other Standards
(1)	(2)	(3)	(4)	(5)
i)	*D*-glucose content (on dry basis), percent by mass, *Min*	99.5	—	Appendix B of IS 873 : 1974
ii)	Total solids content, percent by mass, *Min*	90	E	—
iii)	Ash, sulphated (on dry basis), percent by mass, *Max*	0.1	—	7 of IS 4706 (Part 2) : 1978
iv)	Acidity	To satisfy the test	F	—
v)	Sulphur dioxide, mg/kg, *Max*	70	—	11 of IS 4706 (Part 2) : 1978
vi)	Arsenic (as As), mg/kg, *Max*	1.1	G	—
vii)	Copper (as Cu), mg/kg, *Max*	2	H	—
viii)	Lead (as Pb), mg/kg, *Max*	0.5	J	—
ix)	Zinc, mg/kg, *Max*	50.0	—	15 of IS 6287 : 1985
x)	Tin, mg/kg, *Max*	250.0	—	16 of IS 6287 : 1985
xi)	Mercury, mg/kg, *Max*	1.0	K	—
xii)	Cadmium, mg/kg, *Max*	1.5	L	—

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean, high density polyethylene (HDPE) or polylined high density polywoven bags or biaxially oriented polypropylene (BOPP) or hessian or other textile bags which may be either paper, polyethylene or cotton lined or in suitable multi-walled paper bags or in lined corrugated fibreboard containers or in solid fibreboard containers or in leverlid tin plate containers.

5.2 Marking

5.2.1 Each container shall be suitably marked/printed with indelible insoluble ink so as to give the following information:

a) Name of the material;
b) Indication of source of manufacture;
c) Batch or code number;
d) Date of manufacture;
e) Net mass; and
f) Any other details required under the *Standards of Weights and Measures (Packaged Commodities) Rules, 1977/ Prevention of Food Adulteration Rules.*

5.2.2 The containers may also be marked with Standard Mark.

6 SAMPLING

Representative samples of the material shall be drawn and conformity of the material to the requirements of this specification shall be determined by the method prescribed in Annex M.

7 TESTS

7.1 The tests shall be carried out as prescribed in **4.4**, **4.5**, **4.6** and col 4 and 5 of Table 1.

7.2 Quality of Reagents

Unless specified otherwise, pure chemicals shall be employed in tests and distilled water (*see* IS 1070 : 1977) shall be used where the use of water as a reagent is intended.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
266 : 1977	Sulphuric acid (*second revision*)
323 : 1959	Rectified spirit (*revised*)
873 : 1974	Liquid glucose (*first revision*)
1070 : 1977	Water for general laboratory use (*second revision*)
4706 (Part 2) : 1978	Methods of test for edible starches and starch products: Part 2 Chemical methods (*first revision*)
4905 : 1968	Methods for random sampling
6287 : 1985	Methods for sampling and analysis for sugar confectionery (*first revision*)

ANNEX B

(*Clause* 4.4)

DETERMINATION OF LOSS ON DRYING

B-1 APPARATUS

B-1.1 Moisture Dish

Made of aluminium sheet of thickness 0·50 mm, 55 mm in diameter and 15 mm in height with a tight-fitting cover. The dish shall have a polished surface.

B-2 PROCEDURE

B-2.1 Weigh accurately about 5 g of the material into the tared moisture dish fitted with the cover. Uncover the dish and dry the material in an oven at 105°C for 4 hours. Replace the cover of the dish, cool in a desiccator and weigh. Repeat drying the material in the dish at hourly intervals, cooling and weighing until the loss in mass between two successive weighings is less than one milligram. Record the lowest mass

B-3 CALCULATION

B-3.1 Loss on drying, percent by mass $= \dfrac{100\,(M_1 - M_2)}{(M_1 - M)}$

where

M_1 = mass, in g, of the dish with the material before drying,

M_2 = mass, in g, of the dish with the material after drying, and

M = mass, in g, of the empty dish.

ANNEX C

(*Clause* 4.5)

DETERMINATION OF SPECIFIC ROTATION

C-1 REAGENT

C-1.1 Ammonium Hydroxide Solution — rd 0.90

C-2 PROCEDURE

C-2.1 Preparation of Solution of the Material

Dry a convenient quantity of the material at 105°C for 6 hours in an air-oven and cool it in a desiccator Weigh accurately 10 g of the dried material into a 100-ml volumetric flask, dissolve in a small quantity of water, add 0.2 ml of ammonium hydroxide solution, make up the volume with water and mix.

C-2.2 Determine the optical rotation of the solution within 30 minutes from the time it was dissolved using a suitable type of polarimeter and sodium light for all observations.

C-3 CALCULATION

C-3.1 Specific rotation of the material $= \dfrac{100\,\alpha}{l \times c}$

where

α = optical rotation in degrees,

l = thickness, in decimetre, of the layer examined, and

c = mass, in g, of the substance contained in 100 ml of solution at 25°C

ANNEX D

(*Clause* 4.6)

DETERMINATION OF SPECIFIC VOLUME

D-1 APPARATUS

D-1.1 Cylinder Funnel Assembly

Place a 250 ml graduated cylinder, having a graduated section 24 to 26 cm in length, on a horizontal surface Suspend a funnel, having a stem 30 mm in length and 70 mm outside diameter in a vertical position by means of a ring support on a ring stand, with stem centred inside cylinder 6 cm above 250 — ml mark.

NOTE — If cylinder and funnel of above dimensions are not available, use any 250 ml cylinder and glass funnel having dimensions nearest to above and keep the same assembly for different determinations

D-1.2 Balance

Suitable torsion or top loading balance. Capacity 2 kg with 0·1 g accuracy.

D-2 PROCEDURE

D-2.1 Accurately weigh about 100 g of the sample and with the aid of a spoon or spatula, carefully add sample to the funnel until the complete sample is transferred in the cylinder. Level the top surface of material in the cylinder by tilting the cylinder, if necessary. Read volume in ml of the material from graduations on the cylinder.

D-3 CALCULATION

Specific volume, ml/100 g $= \dfrac{V}{M} \times 100$

where

V = volume of sample observed (*see* **D-2.1**); and

M = mass of sample, in g, taken for the test

ANNEX E

[*Table* 1, *Sl No.* (ii)]

DETERMINATION OF TOTAL SOLIDS CONTENT

E-1 The total solids can be calculated as follows after determining the loss on drying (*see* **B-3.1**).

Total solids content, percent, *m/m* = 100 − loss on drying (**B-3.1**).

ANNEX F

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF ACIDITY

F-1 REAGENTS

F-1.1 Phenolphthalein Indicator Solution

0·2 percent (*m/v*) Dissolve 0·2 g of phenolphthalein in 60 ml of rectified spirit (*see* IS 323 : 1959) and add sufficient quantity of water to produce 100 ml.

F-1.2 Standard Sodium Hydroxide Solution — 0·02 N

F-2 PROCEDURE

F-2.1 Weigh accurately about 5 g of the material into a conical flask and dissolve it in 50 ml of water free from carbon dioxide gas. Titrate the content of the conical flask with standard sodium hydroxide solution using phenolphthalein solution as indicator

F-3 CALCULATION

F-3.1 Acidity, as ml of 0·02 N standard sodium hydroxide solution required to neutralize 5 g of the material

$$= \frac{250\,VN}{M}$$

where

V = volume, in ml, of standard sodium hydroxide solution required to neutralize the quantity of the material taken for the test,

N = normality of standard sodium hydroxide solution; and

M = mass, in g, of the material taken for the test.

F-4 REPORT

F-4.1 The material shall be deemed to have satisfied the requirements of the test if the acidity, expressed as volume of 0·02 N standard sodium hydroxide solution required to neutralize 5 g of the material, does not exceed 1·0 ml under the conditions of the test

ANNEX G

[*Table* 1, *Sl No.* (vi)]

TEST FOR ARSENIC

G-1 The method as given in Appendix D of IS 873 : 1974 shall be adopted with the following modification

In **D-3.1** instead of 'brominated hydrochloric acid' use 'stannated hydrochloric acid'.

ANNEX H

[*Table* 1, *Sl No.* (vii)]

TEST FOR COPPER

H-1 The method as given in Appendix E of IS 873 : 1974 shall be adopted with the following modification

In **F-2.1** instead of '30 ml standard solution' use '2 ml standard solution'.

ANNEX J

[*Table* 1, *Sl No.* (viii)]

TEST FOR LEAD

J-1 The method as given in Appendix F of IS 873 : 1974 shall be adopted with the following modification:

In **F-2.1** instead of '5·0 ml of dilute lead solution' use '0·5 ml dilute lead solution'.

ANNEX K

[*Table* 1, *Sl No.* (xi)]

TEST FOR MERCURY

K-1 PRINCIPLE

K-1.1 Sample is digested with nitric acid (HNO_3) and sulphuric acid (H_2SO_4) under reflux in a special apparatus. Mercury is isolated by dithizone extraction, copper is removed and mercury is estimated by photometric measurement of mercury dithizonate.

K-2 APPARATUS

K-2.1 Special Digestion Apparatus

(*see* Fig. 1). The apparatus is made from pyrex glass with standard taper joints throughout. Unit A is a modified Soxhlet extractor, of 5 cm outer diameter and 200 ml capacity, without an inner siphon tube but equipped with a stopcock on the tube leading to digestion flask, *D*. With the stopcock open, the apparatus is in reflux position; when closed, the unit serves as a trap for condensed water and acids. The top of *A* is attached to a Friedrich's condenser, 35 cm long. The bottom of *A* is attached to the centre neck of a 2-neck standard taper 24/40 round-bottom 500-ml flask, *D*. Necks are 3 cm apart to provide clearance. The second neck is used for attaching a 75-ml dropping funnel *B*.

K-3 REAGENTS

K-3.1 Mercury Standard Solution

K-3.1.1 *Stock Solution*

1 mg/ml; prepared from dry, recrystallized mercuric chloride ($HgCl_2$) (67·7 mg/50 ml).

K-3.1.2 *Working Solution*

2 μg/ml is convenient. Prepare from stock solution and store in pyrex bottles. Add hydrochloric acid (HCl) in a proportion of 8 ml/litre to all standards before diluting to final volume.

K-3.2 Chloroform ($CHCl_3$)

Distil from hot water-bath, collecting distillate in absolute alcohol in the proportion of 10 ml alcohol to 1 litre distillate. Intermittently shake receiver during distillation.

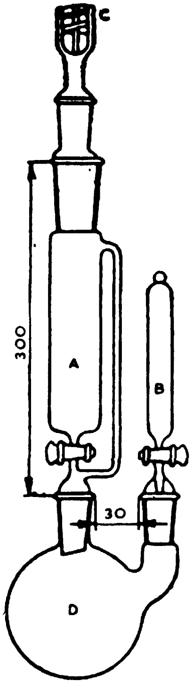

FIG. 1 SPECIAL DIGESTION APPARATUS FOR DETERMINATION OF MERCURY RESIDUES

K-3.3 Dithizone Solution

Dissolve about 1 g commercial reagent in 50-75 ml chloroform and filter if insoluble material remains. Extract in a separator with four 100 ml portions of metal-free (redistilled) NH_4OH (1:99) (dithizone passes into the aqueous phase to give an orange solution). Filter aqueous extracts into a large seaprator through a cotton plug inserted in the stem of the funnel. Acidify slightly with dilute HCl and extract dithizone with two or three 20 ml portions of $CHCl_3$. Combine extracts in separator and wash 2 or 3 times with water. Drain $CHCl_3$ into a beaker and evaporate with gentle heating on a steam bath, avoiding sputtering as the solution goes to dryness. Remove last traces of moisture by heating for 1 hour at about 50°C in vacuum. Store dry reagent in the dark in a tightly stoppered bottle. Prepare stock solution in redistilled $CHCl_3$ (100 mg/litre is convenient) and store in a refrigerator. Prepare dilutions as needed.

K-3.4 Sodium Thiosulphate Solution

1·5 percent prepared daily.

K-3.5 Sodium Hypochlorite Solution (NaOCl)

Preferably 5 percent available chlorine reagent. The reagent varies in available chlorine content. Determine the strength. Store in refrigerator when not in use and determine titer monthly. (Certain preparations of hypochlorite intended for household use contain traces of Hg. If these preparations are used, determine blank. Reagent containing more than 0·1 μg Hg/ml should not be used.)

K-3.6 Dilute Acetic Acid

30 percent (*v/v*)

K-3.7 Hydroxylamine Hydrochloride Solution ($H_2NOH.HCl$)

20 percent (*m/v*). Extract with dilute dithizone until $CHCl_3$ layer remains green, remove excess dithizone with $CHCl_3$ and filter.

K-4 PREPARATION OF SAMPLE

K-4.1 Place weighed sample in digestion flask with 6 glass beads. Add 20 ml HNO_3 to the sample, swirl flask, and let it stand for half an hour in digestion assembly before heating. Add 25 ml water and heat cautiously with small rotating flame until the initial vigorous reaction is over and foaming ceases. [Original reaction must not proceed violently or the evolved nitrogen peroxide (NO_2) will carry vapours of digest mechanically through the condenser and cause loss of Hg]. After initial reaction is complete, apply heat so that digest just refluxes. If the mixture darkens, add NHO_3 dropwise through the funnel as needed. Continue refluxing for half an hour or until the digest does not change consistency, and cool.

K-4.2 Slowly add 20 ml of cold nitric acid — sulphuric acid mixture (1:1) [Use 100 ml of acid mixture for about 5 g (dry mass) of sample]. Heat with a small flame, subsequently adding nitric acid dropwise as needed to dispel darkening of the digest. Continue heating until the fibrous material is apparently digested. Turn the stopcock of the soxhlet unit to trap water and acids, and continue heating. Let the digest become dark brown (not black) before adding further increments of nitric acid. (Fats and waxes cannot be totally digested by hot acids under reflux. Therefore, no attempt should be made to effect complete digestion in this step.) When all except fat and wax is in solution, let digest cool, and cautiously drain water and acids into main digest. Cool and pour two 25-ml portions of water through condenser and intermediate unit. Remove reaction flask, chill under cold water or by surrounding with ice to solidify fats and waxes, and filter off insoluble matter on small plug of glass wool. Rinse reaction flask and filter pad successively with two 10-ml portions of water. Remove soxhlet unit and wash it and flask with hot water to remove insoluble material. Pour hot water through condenser to remove volatile fats and oils. Discard all washings.

K-4.3 Connect flask containing filtered sample solution to assembled apparatus, heat and collect water and acids in trap. Complete digestions, using small additions of nitric acid as needed. In final stages of digestion, adjust flame until digest reaches incipient boiling (solution simmers) and acid vapours do not rise beyond lower half of condenser. Continue heating 15 minutes after last addition of nitric acid. Digest should now be colourless or pale yellow. Let digest cool, drain trapped liquids carefully into reaction flask, and add two 50-ml portions of water through condenser. Reflux solution until all NO_2 is expelled from apparatus. Add 5 ml 40 percent *m/v* urea solution and reflux for 15 minutes. Digest should be colourless or pale yellow.

K-5 ISOLATION OF MERCURY

K-5.1 Titrate 1 ml of prepared sample solution (**K-4.3**) with standard alkali. Add a calculated amount of concentrated ammonia to reduce acidity to 1·0 N; swirl flask during addition of the ammonia to avoid local excess (solution should never be ammoniacal to avoid formation of mercury complexes).

K-5.2 Transfer sample solution to a 500-ml separator. Add 10 ml of 4 mg/litre dithizone and shake vigorously for 1 minute. (If the characteristic green colour of dithizone is visible in the chloroform layer, indicating excess of dithizone, the amount of mercury is within 0·5 μg.) Let the layers separate and drain chloroform layers quickly to a second separator containing 25 ml of 0·1 N Hydrochloric Acid and 5 ml $H_2NOH.HCl$ solution. (Small amount of oxidizing material may still be present. On long contact with dithizone solution, oxidizing substances may destroy dithizone reagent and prevent extraction of mercury.)

K-5.3 Repeat extraction of sample solution with two 5-ml portions of dithizone solution, transferring $CHCl_3$ layer successively to second separator If first extraction indicates more than 5 μg Hg, add stronger concentrations of dithizone, as indicated by table under **K-7.1**, until after 1 minute of vigorous shaking, $CHCl_3$ layer contains dithizone in marked excess Drain $CHCl_3$ layer into second separator containing 0 1 N HCl and again extract sample solution 10-ml portions of 4 mg/litre dithizone solution, with two draining each successive extract into second separator

K-5.4 Shake the contents of second separator vigorously for 1 minute and drain $CHCl_3$ layer into a third separator containing 50 ml of 0 1 N HCl (Shaking dithizone extract with dilute acid in second separator removes entrained organic matter With biological materials or those of high protein content, aqueous layer is usually light yellow because of nitrated organic compounds Small amounts are carried into the third separator where they are destroyed by chlorine) Extract solution in second separator with 1-2 ml $CHCl_3$ and transfer organic layer to third separator

K-5.5 To the contents of the third separator add 2 ml sodium thiosulphate solution, shake vigorously for 1 minute, let layers separate, drain off $CHCl_3$ as completely as possible, and discard (Copper if present is removed as dithizonate) Extract again with 1-2 ml $CHCl_3$, drain carefully, and discard Add 3 5 ml NaOCl reagent (or enough solution of different titer to furnish 175 mg of available chlorine) to decompose mercury thiosulphate complex and oxidize excess thiosulphate, and shake vigorously for 1 minute Add 5 ml H_2N OH HCl reagent from pipette, taking care to wet both stopper and neck of separator Shake vigorously for 1 minute Hold the mouth of the separator in front of the air vent and blow out any remaining gaseous chlorine Stopper separator and shake vigorously for 1 minute It is imperative that all hypochlorite be reduced Trace amounts remaining would oxidize dithizone, subsequently added, to a yellow oxidized form which would be measured in a photometer as mercury Extract the solution with 2-3 ml of $CHCl_3$, drain off the organic layer carefully, and discard Final aqueous solution should be colourless

K-6 DETERMINATION

K-6.1 To the third separator, add 3 ml of 30 percent acetic acid and an appropriate volume and concentration of dithizone solution as indicated in the table (*see* **K-7.1**) and proceed with colorimetric determination of mercury as in **K-7**, converting absorbance (*A*) measured at 490 nm to μg mercury from the working curve

K-7 PREPARATION OF STANDARD CURVE

K-7.1 The following table is useful in preparing the standard curve and for establishing appropriate mercury range in sample solutions when 1-cm cell is used

Mercury Range μg	*Dithizone Concentration* (mg/litre)	*Volume Dithizone* (ml)
(1)	(2)	(3)
0— 10	6	5
0— 50	10	25
0—100	10	40

K-7.2 Prepare a working curve of required range, starting with the blank and extending to final standard of the range, with 4 intermediate increments Add appropriate amounts of mercury to 50 ml 0 1 N HCl in a separator Add 5 ml H_2NOH HCl reagent and 5 ml $CHCl_3$ and shake vigorously for 1 minute Let the layers separate, drain off $CHCl_3$ and discard, being careful to remove as completely as possible all droplets of $CHCl_3$ Add 3 ml of 30 percent acetic acid and an appropriate volume of dithizone solution, shake vigorously for one minute, and let the layers separate (acid aids in stablizing mercuric dithizonate) Insert cotton plug into the stem of the separator and collect dithizone extract (discarding the first ml) in test tube for transfer to appropriate cell Make photometer readings at 490 nm (since both dilute dithizone and mercuric dithizonate are somewhat unstable) read immediately Plot *A* against μg mercury

K-8 PRECAUTIONS

K-8.1 The critical step is digestion of the sample, which must be almost complete, otherwise the residual organic matter may combine with mercury and prevent or hinder extraction with dithizone The oxidising material in digest must also be destroyed otherwise the dithizone reagent is decomposed and mercury is not quantitatively extracted Because of volatility of mercury components careful heating of digest during sample preparation is required Acidity of final sample solution (after partial neutralization with ammonia) before extraction should be about 1 N and not more than 1 2 N Do not use silicone grease in stopcocks

ANNEX L

[*Table* 1, *Sl No.* (xii)]

METHOD FOR DETERMINATION OF CADMIUM

L-0 Sample is digested with HNO_3, H_2SO_4, and H_2O_2. All reactive metals are extracted from solution, after adjustment to about *p*H 9, with dithizone-$CHCl_3$. Cadmium is removed by stripping $CHCl_3$ solution with dilute HCl and determined by Atomic Absorption Spectrophotometry at 228·8 nm.

L-1 REAGENTS

L-1.1 Nitric Acid (HNO_3)

Low in lead and cadmium.

L-1.2 Hydrogen Peroxide (H_2O_2) — 50 percent.

L-1.3 Citric Acid

Monohydrate fine crystal.

L-1.4 Thymol Blue Indicator

Triturate 0·1 g indicator in agate mortar with 4·3 ml 0·05 N NaOH. Dilute to 200 ml with H_2O.

L-1.5 Dithizone Solutions

Concentrated solution — 1 mg/ml. Prepare 220 ml in $CHCl_3$. Prepare fresh daily.

L-1.6 Cadmium Standard Solutions

L-1.6.1 *Stock Solution*

1·0 mg/ml. Dissolve 1 000 g cadmium, in 165 ml HCl in 1 l volumetric flask. Dilute to volume with H_2O.

L-1.6.2 *Intermediate Solution*

10 µg/ml. Dilute 10 ml stock solution with 2 N HCl to 1 l. Prepare just before use.

L-1.6.3 *Working Solutions*

Dilute 0, 1, 5, 10 and 20 ml intermediate solution to 100 ml with 2 N HCl (0, 0·1, 0·5. 1·0, and 2·0 µg Cd/ml respectively).

L-2 APPARATUS

L-2.1 Atomic Absorption Spectrophotometer

With hollow-cathode cadmium lamp and 10 cm burner head for air-C_2H_2 flame; wavelength 228·8 nm, range 0-2·0 µg/ml.

L-2.2 Glassware

Thoroughly washed with 8·0 N HNO_3 and rinsed with water. Beakers and watchglasses to be covered during all operations.

L-3 PROCEDURE

L-3.1 Digestion

Weigh 50·0 g sample into 1·5 l beaker. Add several boiling chips or beads, and cover. Carefully add 25 ml HNO_3, cover, and warm gently with flame to initiate reaction. When reaction subsides, add 25 ml HNO_3, warm again, and continue until 100 ml HNO_3 has been added. (Alternatively, add 100 ml HNO_3 all at once, with caution, add let stand at room temperature overnight.) Heat until most nitrous oxide fumes have evolved; control excessive frothing by cooling or quenching with H_2O from wash bottle. Only some cellulose and fatty materials if any, remain undissolved.

To remove any fat visible in hot solution, proceed as follows: Cool beaker in ice, and decant clear, aqueous solution from coagulated oils and solids through glass wool pad into 1 l beaker. Add 100 ml H_2O to 1·5 l beaker with fat, heat, swirl vigorously to rinse fat, chill, and filter as before. Wash funnel and glass wool pad with about 20 ml H_2O.

Add 20 ml H_2SO_4 to sample, dilute to about 300 ml with H_2O, and evaporate over flame until charring begins. When charring becomes extensive, cautiously add 50 percent H_2O_2, 1 ml at a time. Let reaction subside before adding next portion of oxidant, and never add greater than 1 ml at a time. Continue additions of H_2O_2 until solution is colourless. Heat vigorously to SO_3 fumes, adding more H_2O_2 as required to remove char. Heat vigorously to expel excess H_2O_2. Cool colourless digest to room temperature.

Prepare reagent blank of 100 ml HNO_3, 20 ml H_2SO_4, and same amounts of H_2O as added to sample. Cautiously add same amounts of 50 percent H_2O_2, as above, and remove all HNO_3 from blank. Carry out blank through same operations as sample.

L-3.2 Extraction

Add 2 g citric acid to cooled digest and cautiously dilute to about 25 ml with H_2O. Add 1 ml thymol blue indicator and adjust to about *p*H 8·8 by slowly adding NH_4OH while cooling in ice bath, until solution changes from yellowish green to greenish blue. Transfer quantitatively to 250 ml separator, using H_2O, and dilute to about 150 ml.

Cool solution, and extract with two 5-ml portions concentrated dithizone solution, shaking 1-2 minutes each time. Continue extraction with 5 ml portions of dilute dithizone solution until last 5 ml portion of dithizone extract shows no change in colour. Combine dithizone extracts in 125 ml seperator; wash with 50 ml H_2O, and transfer solvent to another 125 ml separator. Extract H_2O, wash with 5 ml $CHCl_3$ and add this to dithizone extracts. Add 50 ml 0 2 N HCl to combined dithizone extracts, shake vigorously for 1 minute, and let layers separate, discard dithizone layer Wash aqueous solution with 5 ml $CHCl_3$ and discard $CHCl_3$. Quantitatively transfer aqueous solution to 400 ml beaker, add boiling chips, and evaporate carefully to dryness Carefully rinse down sides of beaker with 10-20 ml H_2O and again evaporate to dryness

L-3.3 Set instrument to previously established optimum conditions, using air acetylene oxidizing flame and 228 8 nm resonant wavelength. Dissolve dry residue in 5 0 ml 2 N HCl and determine *A* of sample and standard solutions against 2 N HCl as blank Flush burner with H_2O between readings. Use scale expansion controls to obtain 4-10 × expansion, as convenient. Determine cadmium from curve of *A* against μg Cd/ml:

$$\text{ppm Cd} = (\mu\text{g Cd/ml}) \times (\text{ml 2N HCl/g sample})$$

For concentration > 2 0 μg Cd/ml, dilute, solution with 2 N HCl

ANNEX M

(*Clause* 6.1)

SAMPLING OF DEXTROSE MONOHYDRATE

M-1 GENERAL REQUIREMENTS OF SAMPLING

M-1.0 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed

M-1.1 Sampling instrument shall be clean and dry when used.

M-1.2 Precautions shall be taken to draw the samples in a manner so as to protect the samples, the material being sampled, the sampling instruments and the containers for samples from adventitious contamination

M-1.3 The samples shall be placed in clean and dry glass containers.

M-1.4 The sample containers shall be sealed, air-tight after filling and marked with full details of sampling, date of manufacture, name of the manufacturer and other important particulars of the consignment

M-1.5 Samples shall be stored in such a manner that the conditions of storage do not unduly affect the quality of the material.

M-2 SCALE OF SAMPLING

M-2.1 Lot

All the containers in a single consignment of the material drawn from the same batch of manufacturer shall constitute a lot If a consignment is declared to or is known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each groupshall constitute a separate lot.

M-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification

M-2.2 The number *n* of containers to be selected from the lot shall depend on the size of the lot *N* and shall be as given in Table 2.

Table 2 Number of Containers to be Selected for Sampling

Lot Size *N*	No of Containers to be Selected *n*
(1)	(2)
3 to 15	3
16 to 40	4
41 to 65	5
66 to 110	7
Over 110	10

M-2.3 The containers shall be selected from the lot at random In order to ensure the randomness of selection, procedures given in IS 4905 1968 may be followed

M-3 TEST SAMPLES AND REFEREE SAMPLES

M-3.1 Preparation of Individual Samples

In the case of small containers, empty out the contents of each selected container on a sheet of paper and mix thoroughly Cone and quarter as often as necessary till a quantity of the material not less than 500 g is left. In the case large containers, draw with an appropriate sampling instrument small but equal quantities of the material from top, middle and bottom portions of each selected container so as to get a total quantity of the material not less than 500 g From this, take about 250 g of the material and divide it into three equal parts Each part so obtained shall constitute an individual sample representing the container and shall be transferred immediately to thoroughly clean and dry containers, sealed air-tight and labelled with the particulars given under **M-1.4**. The individual samples so obtained shall be divided into three sets in such a way that each set has a sample representing each selected container. One of these set shall be marked for the purchaser, one for the vendor and the third for the referee

M-3.2 Preparation of Composite Sample

Out of the material from each selected container remaining after the individual sample has been taken, equal quantities of the material shall be taken and mixed together so as to form a composite sample weighing not less than 600 g This composite sample shall be divided into three equal parts and transferred to clean dry containers made of glass and labelled with the particulars given in **M-1.4** One of these composite samples shall be for the purchaser, one for the vendor and the third for the referee

M-3 3 Referee Sample

Referee sample shall consist of a set of individual samples (**M-3.1**) and a composite sample (**M-3.2**) marked for this purpose and shall bear the seals of the purchaser and the vendor These shall be kept at a place agreed between the two

M-4 NUMBER OF TESTS

M-4.1 Tests for description, loss on drying, specific volume, D-glucose content, total solids and specific rotation shall be conducted on each of the samples constituting a set of individual test samples (**M-3.1**)

M-4.2 Tests for the remaining characteristics, namely, sulphated ash, acidity, sulphur dioxide, arsenic, lead, copper, zinc, tin, mercury and cadmium, shall be conducted on the composite sample as prepared under **M-3.2**

M-5 CRITERIA FOR CONFORMITY

M-5.1 The lot shall be considered to have satisfied the requirements for description, loss on drying and specific rotation if each of the individual test samples tested as in **M-4.1** satisfies the relevant requirements

M-5.2 The lot shall be considered to have satisfied the requirements for remaining characteristics if all the test results as obtained on the composite sample (**M-4.2**) satisfy the requirements for these characteristics

M-5 3 The lot shall be considered to have satisfied all the requirements of this specification if it passes in **M-5.1** and **M-5.2**

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